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4-(5-Chloro-2-hydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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The title Schiff base compound, $C_{18}H_{16}ClN_3O_2$, has been synthesized by the reaction of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one and 5-chloro-2-hydroxybenzaldehyde in MeOH solution. As expected, the compound adopts an *E* configuration about the imine C=N bond. The N atom is also involved in an intramolecular O—H···N bond which stabilizes the configuration. Intramolecular hydrogen bonds and intermolecular C—H···O interactions are observed.

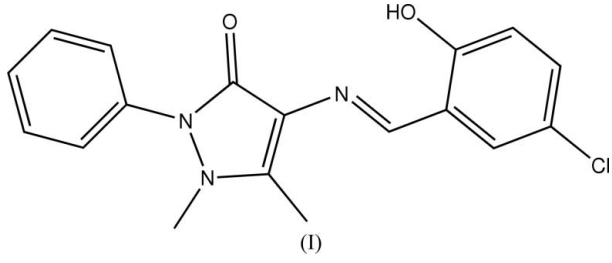
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.064
wR factor = 0.141
Data-to-parameter ratio = 15.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The background to this study is described in a preceding paper (Sun, 2006). As an extension of our work (Sun, Xie *et al.*, 2006; Sun, Zhang, Jin *et al.*, 2006); Sun, Zhang, Wang *et al.*, 2006) on the structural characterization of antipyrine derivatives, a new Schiff base compound, (I), is reported here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. All the bond distances and angles are in normal ranges (Allen *et al., 1987)* and comparable to those observed in similar antipyrine Schiff bases cited above. The C12=N3 bond length of 1.281 (3) Å is as expected for a normal imine double bond. Because of conjugation through this imino double bond, the N1/N2/C7/C8/C9 pyrazoline and C13–C18 benzene rings are nearly coplanar [dihedral angle 1.5 (3)°]. As expected, the

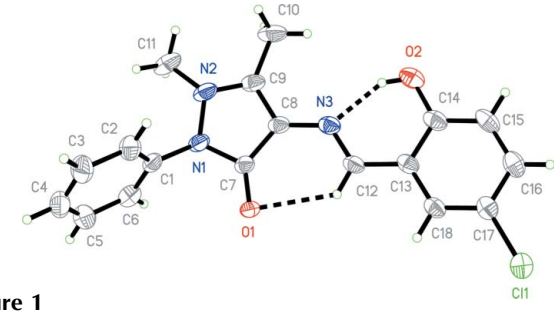


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. The O—H···N and C—H···O hydrogen bonds are shown as dashed lines.

molecular structure of the Schiff base adopts an *E* configuration about the imine C12═N3 bond, as in the other similar antipyrine derivatives that have been reported. In the structure of (I) an intramolecular O—H···N hydrogen bond involving hydroxyl atom O2 and imine atom N3 and another intramolecular C—H···O hydrogen bond involving the C12/H12 group and carbonyl atom O1 stabilize the *E* configuration about the imine C═N bond (Table 1).

A packing diagram of (I) is shown in Fig. 2. In the crystal structure, molecules interact through intermolecular C—H···O hydrogen bonds.

Experimental

All the chemicals were obtained from commercial sources and used without purification. 4-Amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (0.2 mmol, 40.6 mg) and an equimolar quantity of 5-chloro-2-hydroxybenzaldehyde (0.2 mmol, 31.3 mg) were dissolved in methanol (20 ml). The mixture was stirred for 30 min at room temperature to give a clear yellow solution. This solution was kept in air for 8 d, after which time yellow needle-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the methanol (yield 93.6%). Analysis calculated for $C_{18}H_{16}ClN_3O_2$: C 63.25, H 4.72, N 12.29%; found: C 63.13, H 4.74, N 12.23%.

Crystal data

$C_{18}H_{16}ClN_3O_2$
M_r = 341.79
Monoclinic, $P2_1/c$
a = 4.948 (1) Å
b = 23.931 (2) Å
c = 14.294 (1) Å
β = 99.107 (1)°
V = 1671.3 (2) Å^3
Z = 4
D_x = 1.358 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 295 (2) K
Needle, yellow
0.45 × 0.11 × 0.01 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.967, T_{max} = 0.998
13419 measured reflections
3447 independent reflections
2333 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.141
S = 1.09
3447 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 0.7061P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N3	0.82	1.86	2.589 (3)	148
C12—H12···O1	0.93	2.40	3.067 (3)	128
C6—H6···O1^{i}	0.93	2.55	3.425 (4)	156
C10—H10C···O1^{ii}	0.96	2.51	3.467 (4)	172
C15—H15···O2^{iii}	0.93	2.53	3.435 (4)	163

Symmetry codes: (i) $x-1, y, z$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x+1, -y+1, -z$.

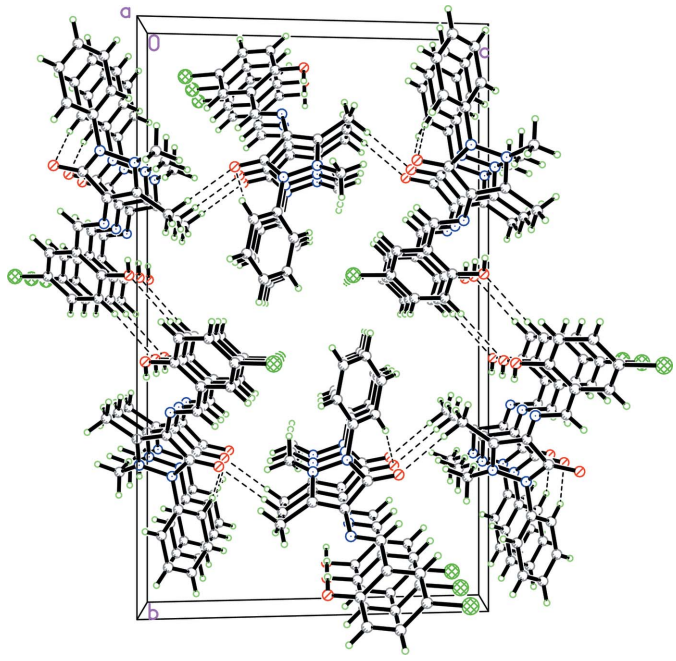


Figure 2
The crystal packing of (I), viewed down the *a* axis. All O—H···N and C—H···O hydrogen bonds are shown as dashed lines.

All H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93 or 0.96 Å) and constrained to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(O), U_{iso}(H) = 1.2U_{eq}(C) for Csp^2 H atoms or U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2002); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2002). *SMART* (Version 5.628), *SAINT-Plus* (Version 6.22) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97 and SHELXL97*. University of Göttingen, Germany.
Sun, Y.-X. (2006). *Acta Cryst.* E**62**, o5845–o5847.
Sun, Y.-X., Xie, X.-H., Zhang, R., Zhang, L.-F. & Xu, L.-X. (2006). *Acta Cryst.* E**62**, o4758–o4760.
Sun, Y.-X., Zhang, R., Jin, Q.-M., Zhi, X.-J. & Lü, X.-M. (2006). *Acta Cryst.* C**62**, o467–o469.
Sun, Y.-X., Zhang, R., Wang, B.-L., Ding, D.-J. & Liu, S. (2006). *Acta Cryst.* E**62**, o4613–o4615.